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Tempering stability of retained austenite in nanostructured dual-phase steels

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Dear Editor and Reviewers,

The authors are very grateful for your very useful comments and suggestions. All the revisions and corrections are highlighted in red color. The responses to your comments are as follows:

1. We have modified the experimental section, including the heat treatment process, the preparation of XRD measuring samples and the calculation of the volume fraction and carbon concentration of retained austenite.
2. Some latest work on Q & P of stainless steels has been cited in the discussion.
3. Six references have been cited and added in the reference list. The sequence of references has been accordingly revised.

The revised manuscript has been re-submitted to your journal.

Sincerely yours,

F. Hu, K.M. Wu, T.P. Hou, A.A. Shirzadi

Tempering stability of retained austenite in nanostructured dual-phase steels

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Abstract

The tempering resistance and stability of retained austenite in super-bainitic and quenching-partitioning martensitic steels were investigated over the temperature range of 400 to 700 °C. The X-ray diffraction analysis and hardness tests showed that the quenching-partitioning-martensitic steel contained a considerable amount of retained austenite (26.6 vol%) and had a relatively high hardness up to 556 HV1 after tempering at about 600 °C. In contrast, the fraction of retained austenite and hardness of super-bainitic steel were considerably lower (24.5 vol% and 385 HV1) after the same tempering cycle. The work also showed that the quenching-partitioning steel

had a higher tempering stability, probably, due to the higher fraction of carbon-rich retained austenite.

Keywords: Super-bainitic steels, quenching-partitioning martensitic steels, tempering, Microstructure.

Introduction

Super-bainitic steels, consisting of bainitic ferrite plates and film-like retained austenite, have recently been developed by Bhadeshia et. al. ¹⁻⁴. These nanostructured steels are the product of bainitic transformation at relatively low temperatures (e.g. 200 to 300 °C). These steels offer a desirable combination of high ultimate strength (between 1.77 to 2.2 GPa), ductility (~30 %) and fracture toughness (~45 MPa m^{0.5}). The mechanical properties of super-bainitic steels mainly depend on the size and shape of bainitic ferrite plates (20-65 nm in thickness) and amount of retained austenite ^{2, 5}. High ductility and toughness of these steels are attributed to their high volume fraction of retained austenite which is usually more than 20 vol% ⁵⁻⁶. However, the heat treatment for bainitic steels usually takes up to several days even if Co and Al are added into the steels to accelerate the austenite to bainitic transformation ⁷⁻⁸.

Quenching-partitioning martensitic steels were initially developed by Speer et. al. ⁹⁻¹². The quenching-partitioning treatment involves quenching the fully austenitised steel to a temperature between its martensite start (M_s) and finish (M_f) temperatures, followed by an isothermal cycle (*i.e.* partitioning stage) to allow diffusion of carbon

from the martensite to untransformed austenite ⁹. These steels exhibit higher strength and ductility than those of the conventional martensitic steels with the same composition ¹³. This is because more retained austenite can be obtained by quenching-partitioning cycle than by direct quenching cycle [9]. Nevertheless, the volume fraction of retained austenite is usually less than 10 vol% in low or/and medium carbon Q&P steels ¹⁴.

The common feature of super-bainitic and quenching-partitioning martensitic steels is the considerable amount of retained austenite which gives these materials excellent ductility. To-date substantial work has been carried out on design and heat treatment of these steels, aiming to understand the formation mechanisms of retained austenite. The objective of this work was to investigate the tempering stability of the retained austenite in a steel in the super-bainitic and quenching-partitioning martensitic states.

Experimental Procedure

The chemical composition of the steel used in this work is given in Table 1. The as-received steel was homogenized at 1200 °C for 2 days in a vacuum furnace followed by furnace cooling to ambient temperature. Regarding to bainitic transformation, specimens were austenitized at 1000 °C for 30 min and then isothermally transformed at 200 °C for 10 days before quenching in water (21 °C). Regarding to martensitic transformation, specimens were austenitized at 1000 °C for 30 min and then quenched and kept in water for 2 min, then was dipped in a molten

salt bath at 450 °C for 60 s before quenching in water for the second time. The M_s and M_f of the martensitic steel were estimated to be 87 °C and -15 °C, respectively ¹⁵. Therefore, martensitic sample was expected to contain some martensite and un-transformed austenite after the first water quench. Both steels were tempered at various temperatures ranging from 400 to 700 °C for 2 hrs. The effect of tempering time was not studied in this work.

The microstructures of samples were examined using optical and scanning electron (SEM, Sirion 200) microscopes, after conducting standard grinding and polishing processes and using 4 vol% nital solution as an etchant.

Two types of specimens were prepared for transmission electron microscopy (TEM, JEM 2010 HT): thin foils and carbon replicas. The thin foils specimens were cut from bulk specimens, and then mechanically ground to about 50 mm thickness. These specimens were further thinned using a twin-jet electro-polisher at 40 V. The electrolyte consisted of 10 vol% perchloric acid and 90 vol% glacial acetic acid. Thus specimens were used to determine the true thickness of the bainitic ferrite and martensite plates, by measuring the mean linear intercept $L = \pi t / 2$ ⁷. The carbon replica specimens with 3 mm diameter were hot-mounted in Bakelite molding powder, and ground with silicon carbide paper down to 2000 grit and then polished using an Al₂O₃ suspension solution to a particle size of 0.5 μm. They were then chemically etched with a solution of 4 vol% nital for a few seconds. A carbon coating of 20~30 nm (brown gold color) was deposited onto the etched surface in a vacuum. This film was then scored with a sharp blade to divide it into several smaller squares (~1 mm²),

followed by etching in a solution of 8 vol% natal to remove the carbon film, which was then washed in methanol and rinsed with distilled water. Finally, these replica films were mounted on copper grids. Thus specimens were needed to identify the type of carbides within a selected area electron diffraction pattern.

Samples of 10 mm × 10 mm × 2 mm were scanned using an X-ray diffractometer (XRD, Xpert Pro MPD), operating at 40 kV and 45 mA, with a Cu K α radiation. Samples were firstly ground and mechanically polished, and then slightly etched with a solution of 4 vol% natal. The 2 θ scanning angles were from 20° to 100°, with a stepping angle of 0.03342°. Finally, the volume fraction of retained austenite was calculated by measuring the integrated intensities of the (111), (200), (220) and (311) in austenite peaks, (110), (002), (112) and (022) in bainite peaks, and finally (110), (200), (211) and (202) in martensite peaks¹⁶⁻¹⁷ and the error in measuring was about 0.015. The carbon concentration in austenite was estimated by using the measured lattice parameters¹⁷⁻¹⁸ and the calculation error was about 0.10 wt%. The XRD and neutron diffraction work published by Bigg et al.¹⁹⁻²⁰ showed that the XRD technique underestimated the volume fraction of austenite due to surface transformation during preparation by grinding even when chemically polished. Therefore, in the present work, the same sample preparation technique for the two heated steels was carried out to minimize the error for comparative purpose.

Each hardness value, reported in this work, is the average of at least ten Vickers tests (1 kg).

Results

Microstructures prior to tempering

The super-bainitic steel which was isothermally treated at 200 °C for 10 days, had very fine bainitic ferrite and retained austenite - see Figures 1a and 1c. The thickness of bainitic ferrite plates was estimated to be about 50 nm. Some Fe-C clusters were also observed within the bainitic ferrite plates.

The quenching-partitioning-martensitic steel which was treated by quenching at 21 °C for 2 min and then partitioning at 450 °C for 60s, contained retained austenite and fine carbides – see Figures 1b and d. The thickness of martensite laths was estimated to be about 100 nm.

Some dispersed retained austenite islands were observed in both steels although those in the martensitic steel were much coarser than those in the super-bainitic steel – see Figure 2.

Post tempering microstructures

There was no obvious change in the microstructure of the super-bainitic steel after tempering at 400 °C for 2 hrs, and its microstructures still consisted of fine bainitic ferrite plates and retained austenite. However, the retained austenite began to decompose into fine carbide and ferrite particles when the tempering temperature was increased to 500 °C – compare Figures 3a and 3b. The microstructure was coarsened when the tempering temperature was increased to 600 °C for 2 hrs. Figure 3c shows that the amount of precipitated carbides was also increased. Further coarsening and

spheroidization of carbides occurred as the tempering temperature increased to 700 °C – see Figure 3d.

Figure 4 shows the microstructures of the quenching-partitioning-martensitic samples after tempering in different temperatures. It is clear that martensite laths and retained austenite are present with no distinguishable carbides when the tempering temperature was between 400 to 600 °C (Figures 4a, b, c). Once the tempering temperature was increased to 700 °C most of the retained austenite was decomposed into fine carbide and ferrite – see Figure 4d.

Hardness and retained austenite content

The volume fraction of retained austenite, after tempering, was measured by X-ray diffraction. High hardness up to 639 HV1 and a substantial amount of retained austenite (29.1 vol%) were found in the super-bainitic steel – see Figure 5a. The hardness was reached 660 HV1 and the retained austenite fraction decreased to 24.3 vol% when the specimens were tempered at 400 °C for 2 hrs. These changes were due to the partial decomposition of retained austenite and precipitation of fine carbides (*i.e.* precipitation strengthening occurred). Increasing the tempering temperature to 500 °C, has a little or no effect on the hardness (636 HV1). When the tempering temperature was further increased to 600 °C, both the hardness and volume fraction of the retained austenite were sharply dropped (385 HV1 & 24.5 vol%) because of ferrite recovery as well as carbide coarsening and spheroidization.

Slightly low hardness (625 HV1) and but higher retained austenite fraction

(37.2 vol%) were observed in the martensitic steel prior to tempering (Fig. 5b), compared with those of the super-bainitic steel. The hardness of quenching-partitioning-martensitic samples was increased up to 680.6 HV1 and the volume fraction of retained austenite was decreased to 26.6 vol% when the tempering temperature was 500 °C. These changes were attributed to the partial decomposition of retained austenite and carbide precipitation strengthening. Increasing the tempering temperature to 600 °C, reduced the hardness (556 HV1). When the tempering temperature further increased to 700 °C, both hardness and retained austenite fraction were sharply dropped (354 HV1 & 22.7 vol%) because of the decomposition of retained austenite into carbide and ferrite.

The carbon content of retained austenite after tempering was determined using an X-ray diffractometer, based on the dependency of lattice parameters on the carbon content – see Fig. 6. The maximum carbon content in the retained austenite was 1.56 and 1.90 wt% for the bainitic and martensitic steels, respectively. The carbon content of austenite was decreased in both steels when the tempering temperature increased, however, the carbon content was much higher in the martensitic specimens. The peak carbon content in the retained austenite in bainitic samples was higher than the bulk carbon concentration when the tempering temperature was 400 °C and 500 °C (1.29 wt% & 1.11 wt% respectively as to the bulk concentration of 0.95 wt%). However, in the martensitic specimen, the carbon concentration in the retained austenite was still higher than the bulk carbon concentration (1.17 wt% as to the bulk concentration of 0.95 wt%) when tempering temperature reached up to 600 °C.

Identification of carbides

Expectedly, the higher the tempering temperature the higher the amount of carbide precipitates and also the coarser the precipitates. The carbides were identified to be M_2C , M_3C , M_7C_3 and $M_{23}C_6$ type in the samples tempered at 500 °C for 2 h in martensitic steel – see Figure 7. As the tempering temperature was increased to 700 °C, the majority of the carbides proved to be M_3C type in bainitic steel– see Figure 8.

Discussion

Transformation kinetics

The calculated martensite start & finish temperatures (M_s , M_f)¹⁵ of the dual-phase steels used in this work and also the corresponding TTT diagram, estimated using MUCG 83.Mod program²¹, are shown in Fig. 9a. The upper C-curve represents the onset of reconstructive transformations such as the formation of allotriomorphic ferrite and pearlite; whereas the lower curve is for bainitic transformation, and horizontal lines represent the martensite start and finish temperatures⁶. In the super-bainitic steel, the retained austenite fraction at the bainitic transformation temperature (200 °C) can be estimated using equation (1)²²:

$$V_\gamma = \frac{x_\alpha - \bar{x}}{x_\alpha - x_\gamma} \quad (1)$$

where, x_α , x_γ and \bar{x} represent the ferrite carbon content (0.47 wt%), austenite carbon content (1.56 wt%) and average carbon content (0.95 wt%), respectively. The fraction of retained austenite prior to tempering was calculated approximately 44.0

vol% of the bainitic steel. In the martensitic steel, the amount of retained austenite formed after the quenching temperature can be predicted by the Koistinen-Marburger relationship (2) ²³:

$$f_M = 1 - \exp(-0.011 \times \Delta T) = 1 - \exp[-0.011 \times (M_s - Q_T)] \quad (2)$$

where, f_M is the mole fraction of the martensite and ΔT is the temperature difference between M_s (87 °C) and Q_T (21 °C). The fraction of retained austenite prior to partitioning and tempering was calculated approximately 51.6 mol% of the martensitic steel. The volume fraction and carbon concentration of retained austenite strongly depend on the bainite and martensite holding temperature ²⁴. It was also shown that a large amount of retained austenite can be obtained in both nano-structure steels. The carbide precipitation calculation was carried out using J-MatPro program ¹⁵ – see Figure 9b.

Tempering stability of retained austenite

Compared with the bainitic steel, the martensitic steel had much coarser of retained austenite islands, and more volume fraction (37.2 vol%) and carbon concentration of retained austenite (1.90 wt%). Taking the product of retained austenite volume percent with its carbon content ($V_\gamma \times C_\gamma$) as the representation of stability ²⁵, as shown in Fig. 10. The maximum stability of retained austenite, 46.33 and 70.68 vol%·wt%, respectively, were obtained for untempered specimens of the bainitic and martensitic steels. Similarly, for the tempered specimens, the retained austenite in the martensitic steel had higher tempering stability than that in the bainitic

steel. The Q&P treatment enables the enrichment of the retained austenite phase to very high carbon levels. Recent work shows that excessive austenite carbon supersaturation (up to 15 at %) was achieved in austenitic stainless steels ²⁶. In addition, when the tempering temperature increased, the tempering stability of retained austenite was dropped due to the decomposition of some retained austenite.

Conclusions

The effects of tempering temperature (400 to 700 °C) on the microstructure and mechanical properties of super-bainitic and quenching-partitioning-martensitic steels were investigated in this work. The hardness of these nano-structure steels was increased at relatively lower tempering temperatures (400 to 500 °C) due to carbide precipitation. When specimens were tempered at higher temperatures (600 to 700 °C) the reduction in the hardness of martensitic steel was significantly less than that of the super-bainitic steel. The high tempering resistivity/stability of the martensitic steel was attributed to the large amount of carbon-rich retained austenite.

Acknowledgements

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Table 1 Measured chemical composition of steels studied in this work (wt%)

C	Si	Mn	Cr	Mo	Ti	Fe
0.95	0.91	1.30	2.30	0.99	0.17	Bal.

Fig. 1 Optical and TEM micrographs show microstructures of super-bainitic steel (a & c) after transformation at 200 °C for 10 days, and martensitic steel (b & d) after quenching at 21 °C for 2 min and partitioning at 450 °C for 60 s.

Fig. 2 TEM micrographs show retained austenite islands in (a) bainitic and (b) martensitic steels prior to tempering.

Fig. 3 SEM micrographs show microstructure of super-bainitic steel after tempering for 2 h at (a) 400 °C, (b) 500 °C, (c) 600 °C and (d) 700 °C.

Fig. 4 SEM micrographs show microstructure of martensitic steel after tempering for 2 h at (a) 400 °C, (b) 500 °C, (c) 600 °C and (d) 700 °C.

Fig. 5 Measured volume fraction of retained austenite and the hardness in tempered (a) super-bainitic and (b) martensitic steels.

Fig. 6 Carbon content in retained austenite in bainitic and martensitic steels (horizontal line represents the bulk carbon content)

Fig. 7 TEM micrographs show M_3C carbide in martensitic samples after tempering at 500 °C for 2 h, (a) bright field and (b) selected area electron diffraction.

Fig. 8 TEM micrographs show M_3C carbide in super-bainitic samples after tempering at 700 °C for 2 h, (a) bright field and (b) selected area electron diffraction.

Fig. 9 Calculated TTT diagram (a) and wt% of carbides in equilibrium condition as a function of tempering temperature (b).

Fig. 10 Tempering stability of retained austenite in bainitic and martensitic steels studied in this work.

figure 1

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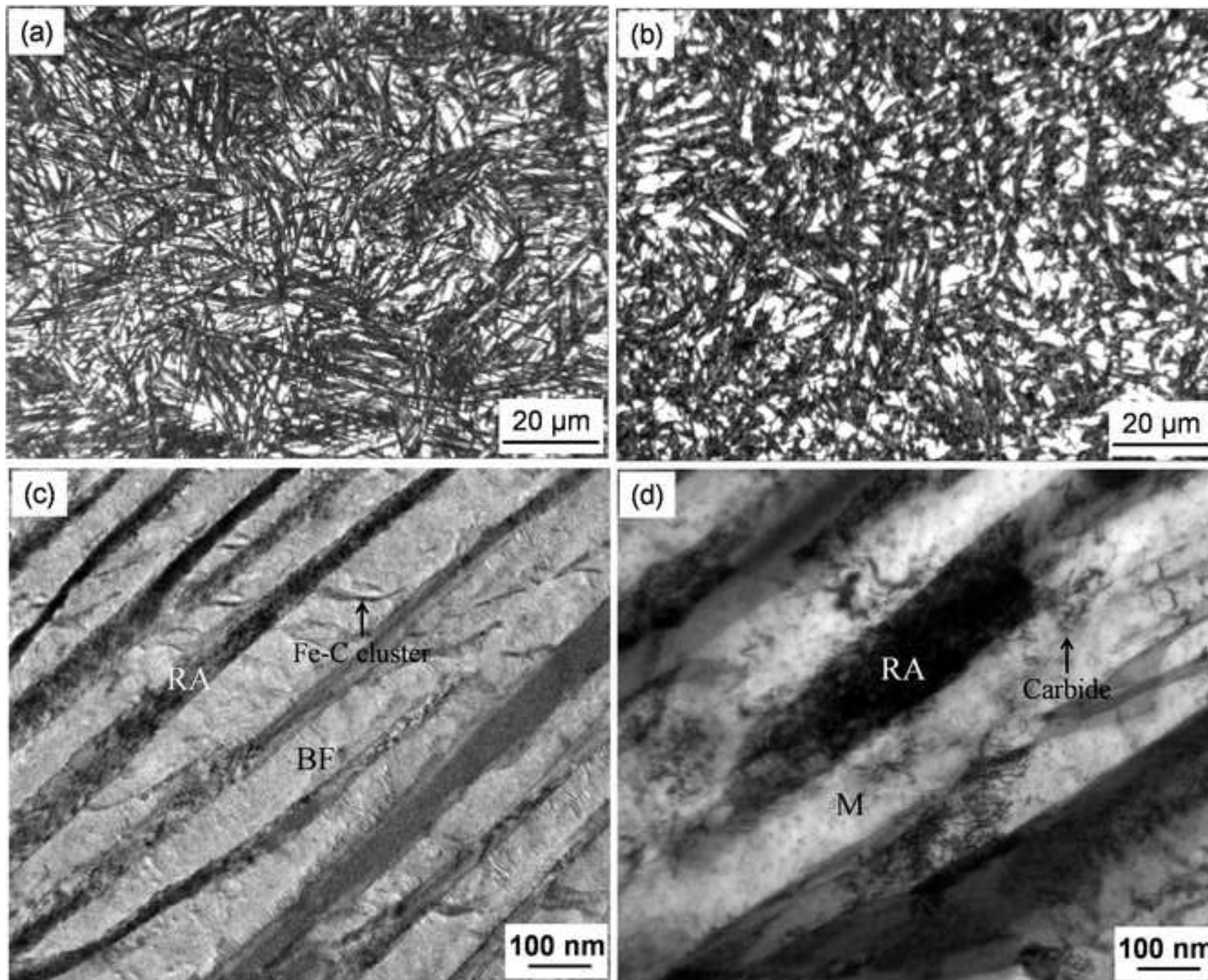


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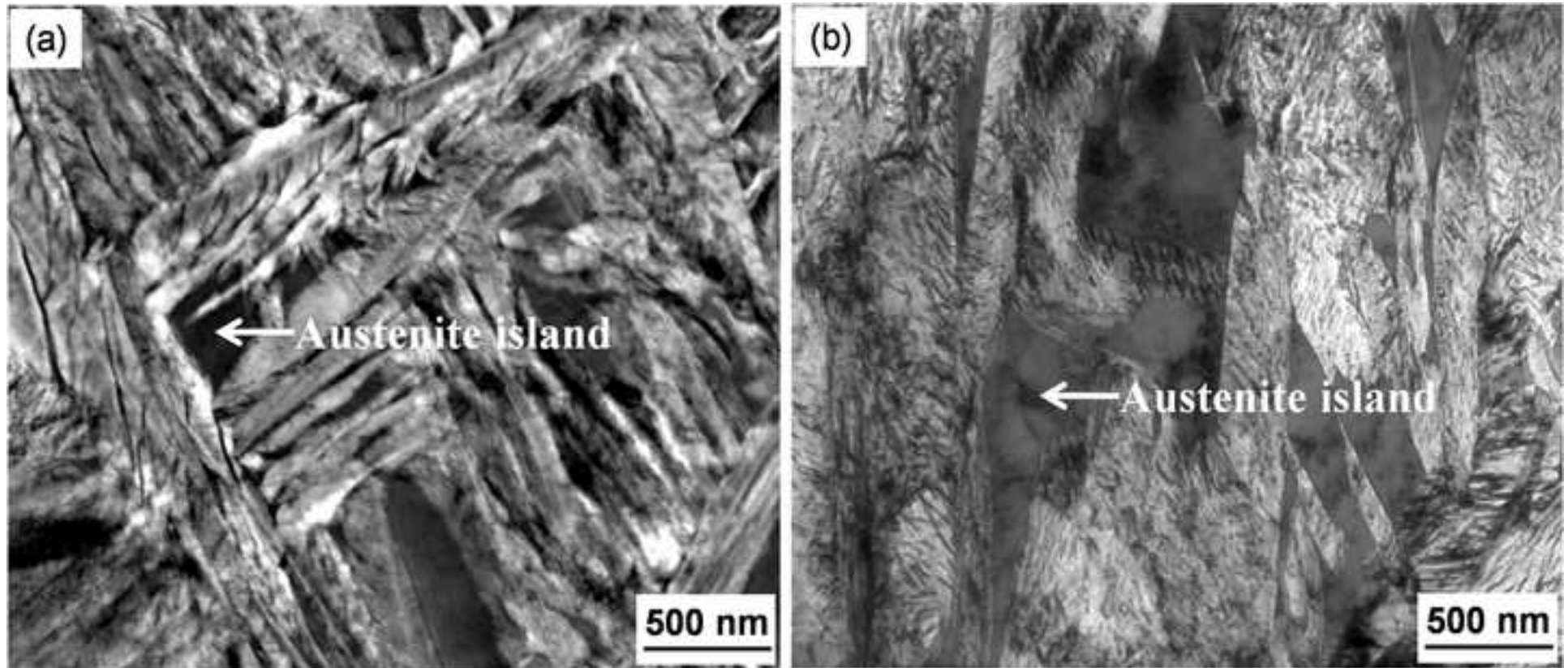


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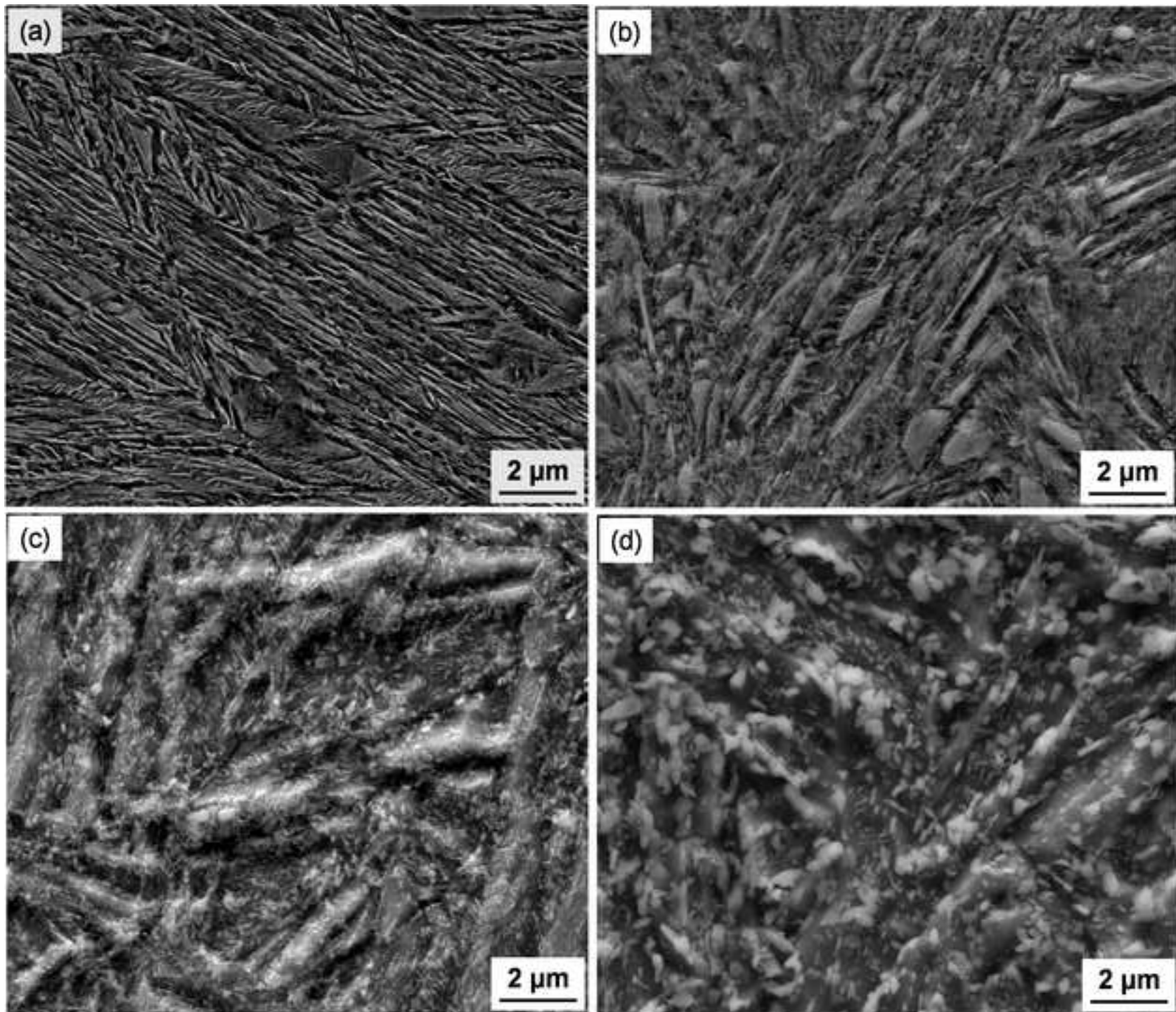


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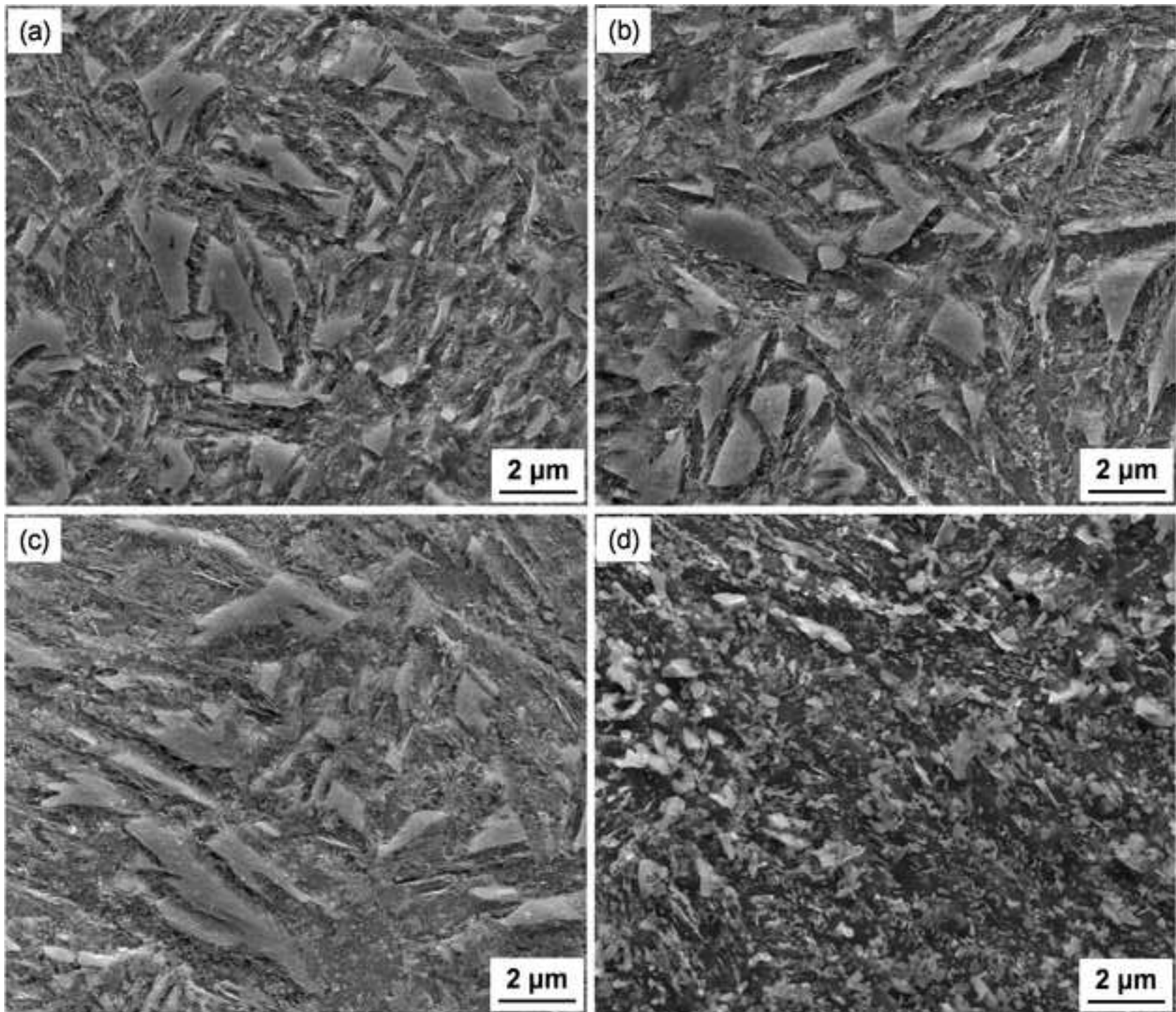


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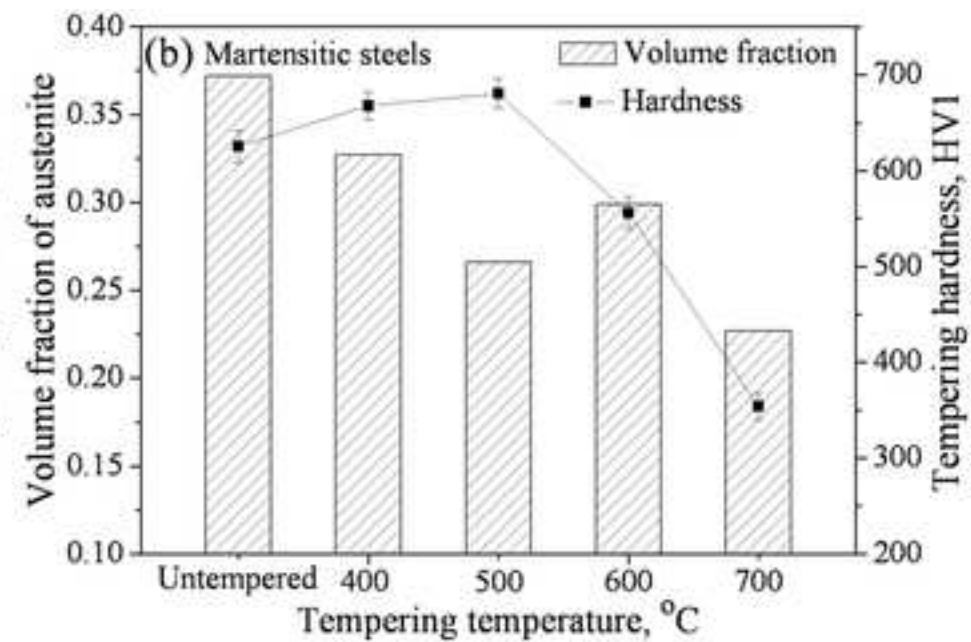
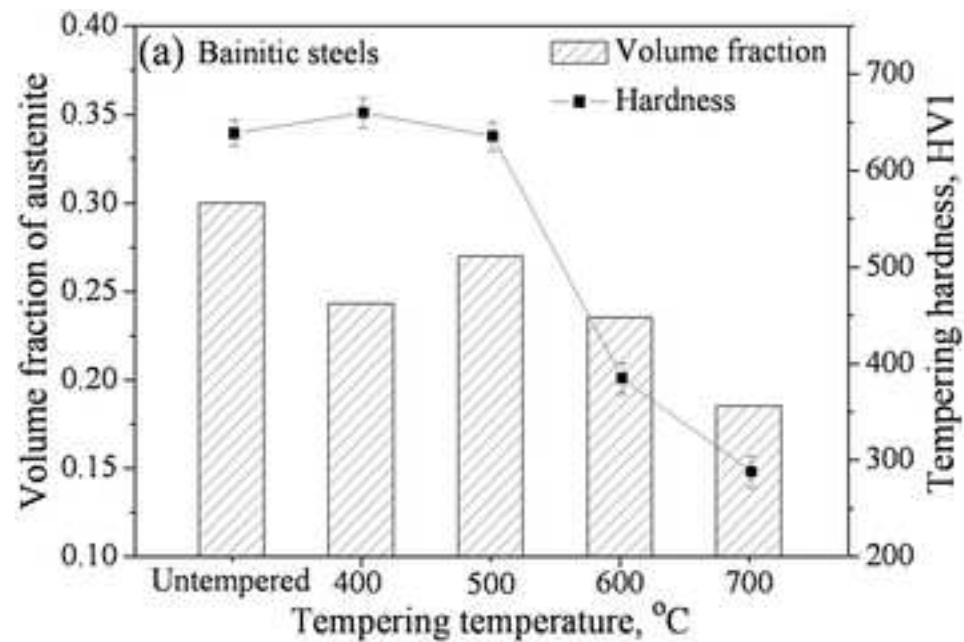


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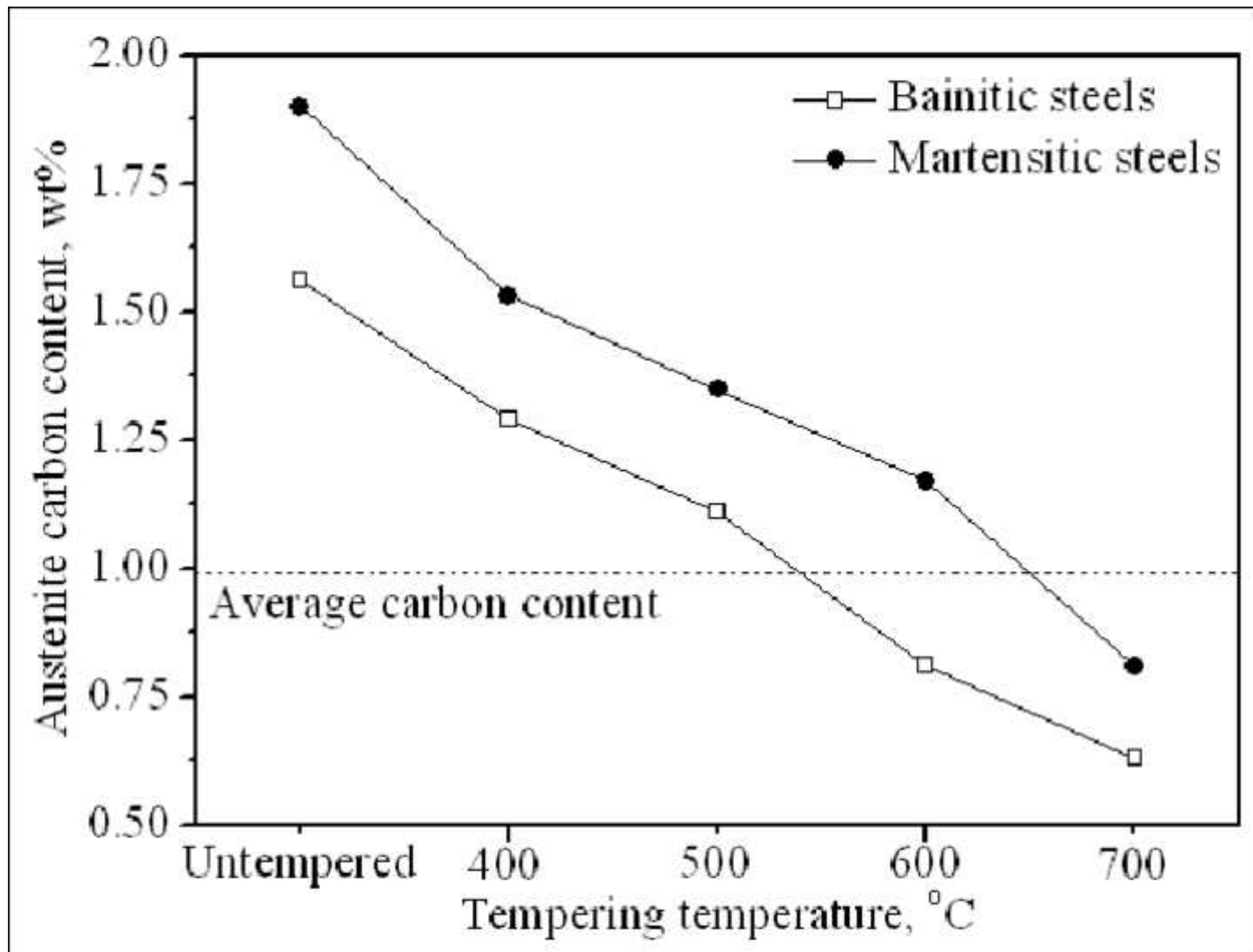


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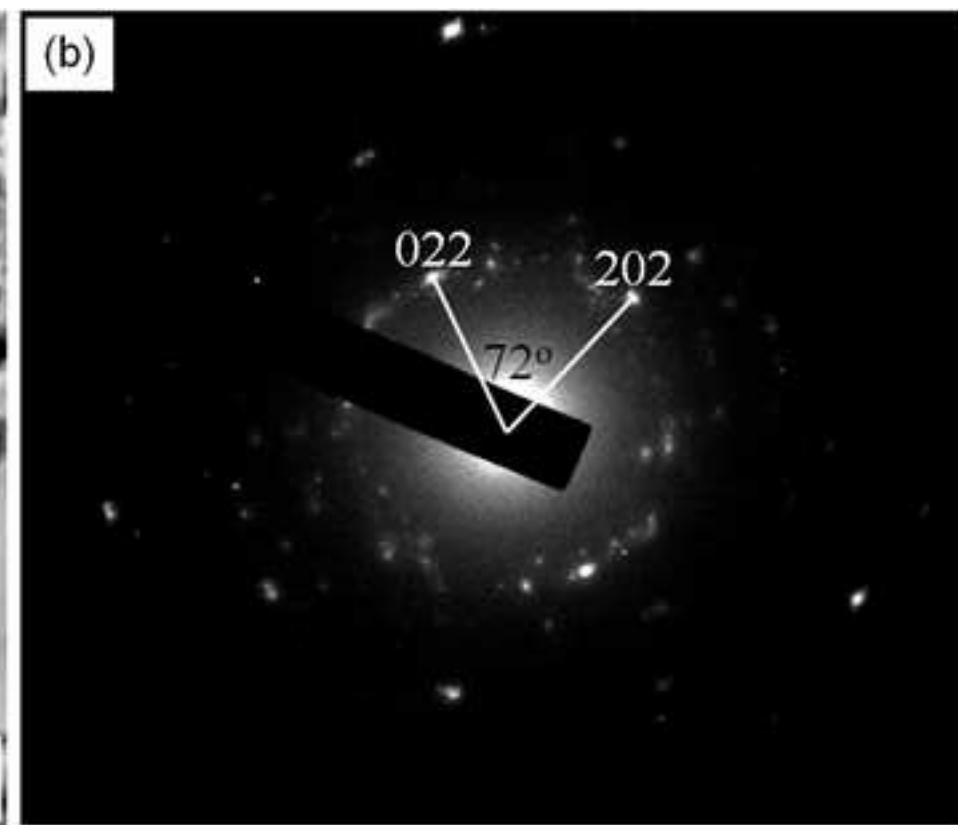
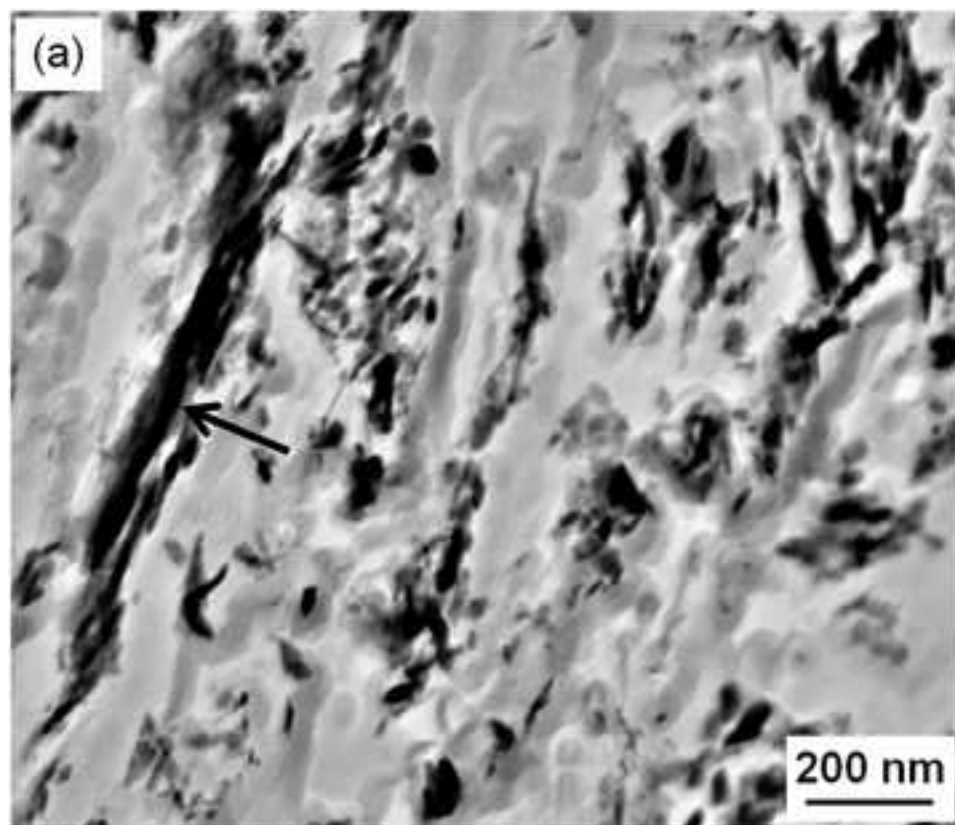


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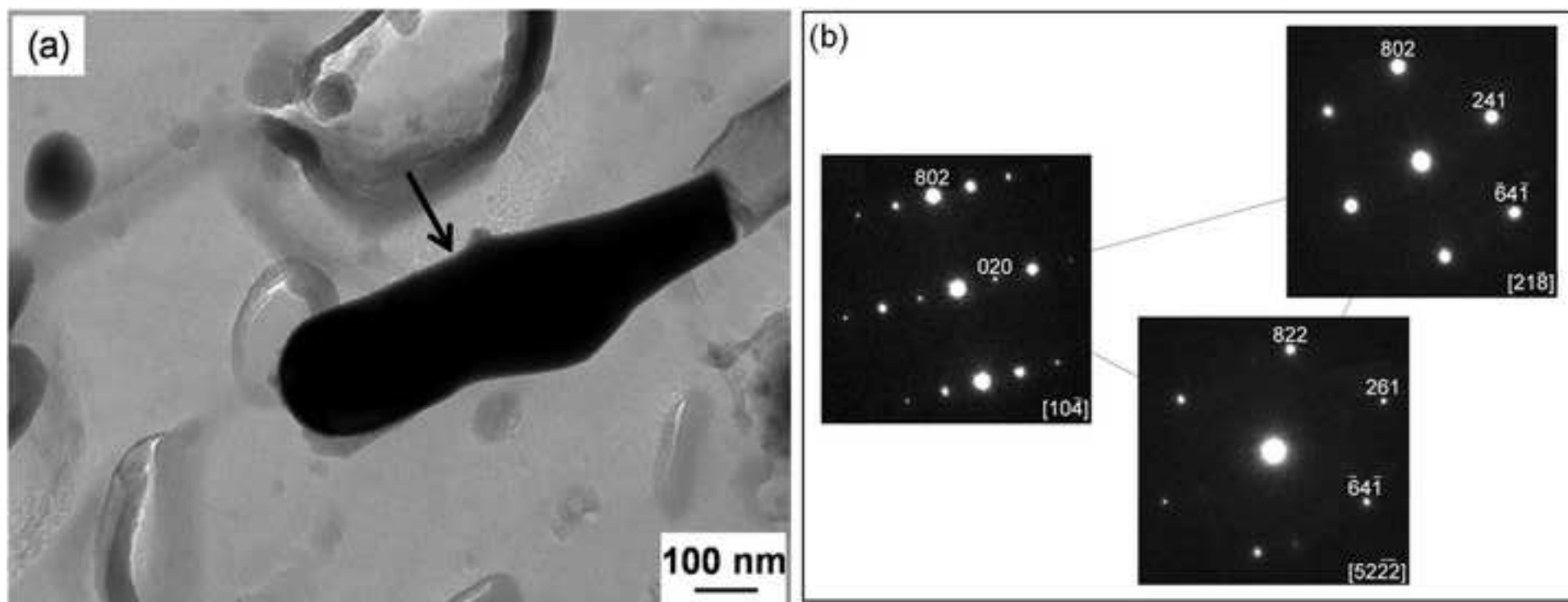


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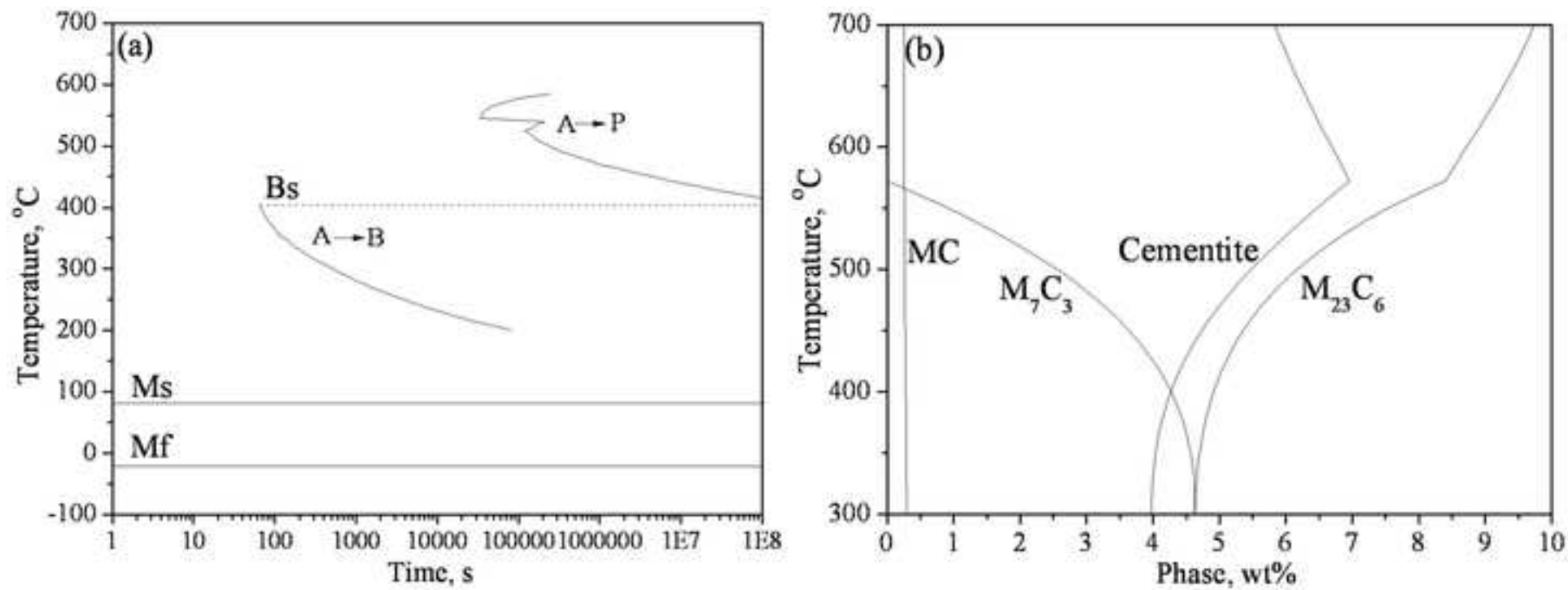
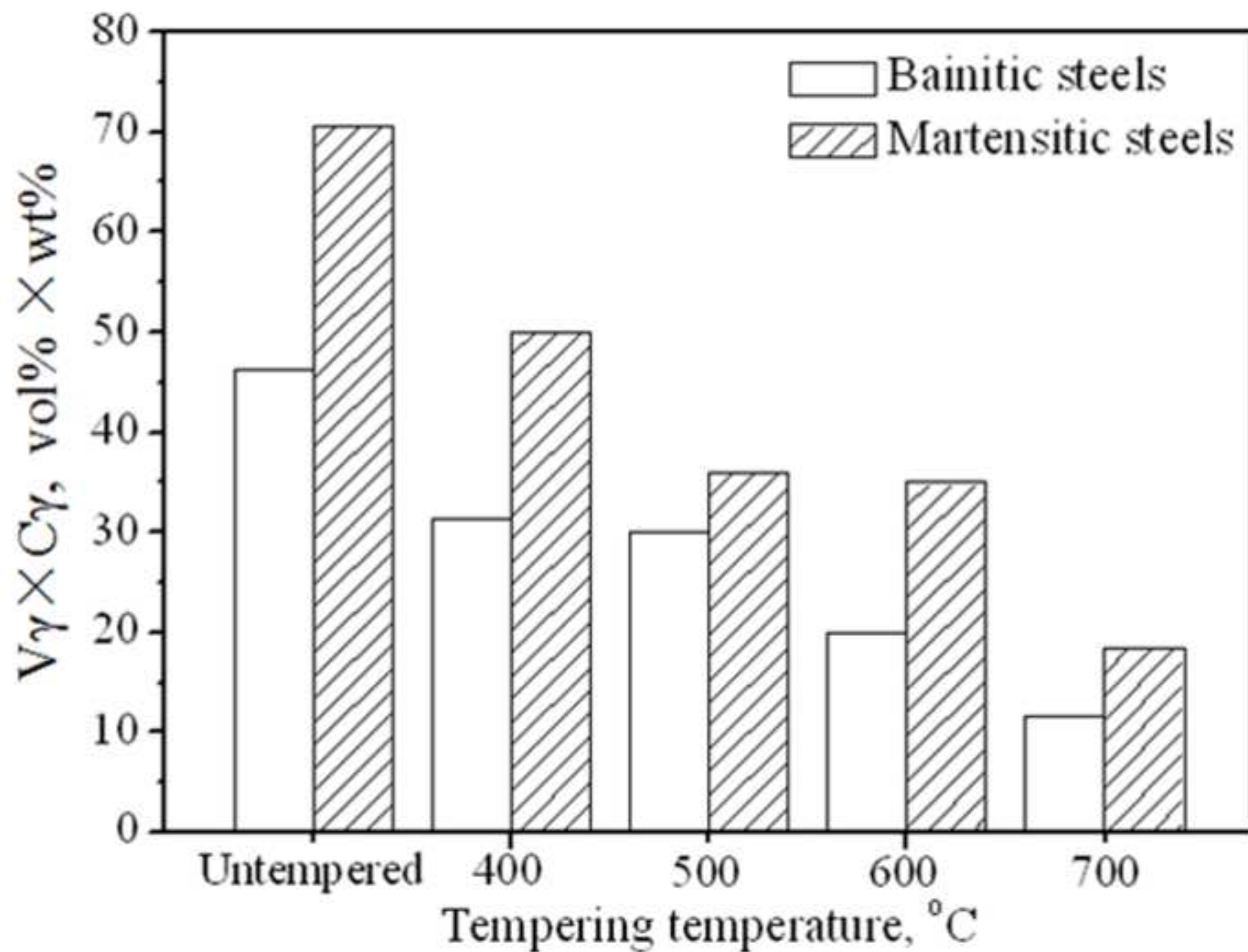


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